IS 8874 (1977): Dimethylamine, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard

SPECIFICATION FOR DIMETHYLAMINE, TECHNICAL

UDC 661·7 : 547·416

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

SPECIFICATION FOR DIMETHYLAMINE, TECHNICAL

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Indian Standard

SPECIFICATION FOR DIMETHYLAMINE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 22 November 1977, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Dimethylamine is used in the manufacture of rayon tyre cord, SBR, acetone formaldehyde resins and nitro-compounds. It is also used in the tanning of leather, insecticides, soil disinfectants and fungicides, drugs and pharmaceuticals, ion-exchange resins, acrylic polishes, resin curing agents, weedicides, drilling of oil wells and in the catalyst for reaction polymerization.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for dimethylamine, technical.

2. TYPES

2.1 The material shall be of the following types:

a) *Type 1* — anhydrous form

b) *Type 2* — 40 percent solution.

3. REQUIREMENTS

3.1 Description

3.1.1 *Type 1* — The material shall mainly consist of dimethylamine (CH_3)$_2$NH and shall be in the form of gas or colourless liquid under pressure and possess a characteristic odour of fish.

*Rules for rounding off numerical values (*revised*).

3.1.2 *Type 2* — The material shall be in the form of liquid and possess a fishy odour.

3.2 Solubility — The material shall be highly soluble in water, and fairly soluble in alcohols and glycols.

3.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR DIMETHYLAMINE, TECHNICAL

(*Clauses* 3.3 and B-5.1)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Appendix A)
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, *Max*	0·5	—	A-2 and A-3
ii)	Ammonia, percent by mass, *Max*	0·01	traces	
iii)	Dimethylamine content, percent by mass, *Min*	99·5	40	
iv)	Monomethylamine, percent by mass, *Max*	0·2	0·1	
v)	Trimethylamine, percent by mass, *Max*	0·2	0·1	

4. PRECAUTIONS IN HANDLING

4.1 The material being flammable and corrosive necessary precautions shall be taken while handling.

5. PACKING AND MARKING

5.1 Packing — The gaseous material shall be filled in gas cylinders under pressure. The material in the solution form shall be packed in mild steel drums.

5.1.1 Necessary safeguard against the risk arising from the storage and handling of this material shall be provided and precautions shall be taken to prevent accident by fire and explosion.

5.1.2 All containers for storage and transport of the material shall, in addition, comply with the requirements of the Red Tariff issued by Indian Railway Conference Association and the requirements laid down from time to time by the Chief Inspector of Explosives, Government of India, for packing, storage and transit of flammable liquids.

5.2 Marking — The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name and type of the material;

b) Net mass of the material;

c) Name of the manufacturer and his recognized trade-mark, if any; and

d) Lot or batch number in code or otherwise.

5.2.1 All the containers in which the material is stored or transported shall be prominently and clearly marked:

**DANGER ! EXTREMELY FLAMMABLE.
HAZARDOUS LIQUID AND VAPOUR UNDER PRESSURE.
LIQUID CAUSES BURNS. VAPOURS EXTREMELY
IRRITATING.**

5.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

6. SAMPLING

6.1 Representative sample of the material shall be drawn and their conformity to the standard shall be judged as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2 and *Table* 1)

METHODS OF TEST FOR DIMETHYLAMINE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water for general laboratory use (*second revision*).

A-2. ANALYSIS OF DIMETHYLAMINE, ANHYDROUS

A-2.0 General — Anhydrous dimethylamine is analysed gas chromatographically by injecting a known volume of the gas and calculating the percentages by mass by the method of area normalization with response factors determined by injecting a standard mixture.

A-2.1 Apparatus

A-2.1.1 *Gas Chromatograph* — with thermal conductivity detectors (hot wire type).

A-2.1.2 *Potentiometric Strip Chart Recorder* — full scale deflection 1 mV.

A-2.1.3 *Column* — of stainless steel or glass, 185 cm long, 4 mm internal diameter and 6 mm external diameter packed with Porapak Q (500-842 microns) coated with 10 percent (*m/m*) of a mixture of 8·9 percent (*m/m*) tetraethylene-pentamine and 1·1 percent (*m/m*) potassium hydroxide.

A-2.1.4 *Syringe* — 2 ml and 10 micro-litre.

A-2.1.5 *Sampling Bomb* — Stainless steel bomb 2·4 metres long and 3·75 cm diameter fitted with needle valves at both ends with 6 mm N.P.T. The bomb should be able to withstand pressure up to 1 765 kN/m^2.

A-2.1.6 *Electric Oven* — Thermostated, fitted inside with a stainless steel coil of 3 mm diameter with ends protruding out through holes on both the side walls of the oven.

A-2.2 Test Substances

A-2.2.1 Ammonia, monomethylamine, diamethylamine, trimethylamine, methanol and water.

A-2.2.2 *Standard Mixture* — A standard mixture of ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water is prepared on *m/m* basis, taking care to see that the total vapour pressure of the mixture does not exceed 98·06 kN.

A-2.3 Procedure

A-2.3.1 *Operating Parameters of Gas Chromatograph*

a) Column oven temperature	90°C isothermal
b) Injection port temperature	150°C
c) Detector block temperature	150°C
d) Carrier gas	Hydrogen with 50 ml/min flow rate
e) Delivery pressure of carrier gas	137·3 kN/m^2
f) Bridge current	200 mA
g) Chart speed	30 cm/h

A-2.3.2 *Test Procedure* — Check and adjust the chromatograph. Inject 1 microlitre of the standard mixture with the help of the hypodermic syringe. By suitably manipulating the attenuator switch, record all the peaks on the chart. Measure the area of all the individual peaks. Calculate the response factors of all the components, considering the factor to be one for monomethylamine.

A-2.3.3 *Sample Injection* — The bomb containing the sample under pressure is connected vertically to one end of the heated coil in the oven with swage lock metallic fittings. The other end (exit end) of the coil is connected with rubber tubing to a bubbler half filled with water. The exit end of the bubbler is again connected with a long rubber tubing which is taken outside the room as a vent. Now the bottom valve of the sample bomb is slowly opened. The sample gets immediately vaporized as it passes through the heated coil kept at 150°C. The vapour coming out through the outlet end of the coil is taken in a 2 ml syringe by piercing the needle through the connecting rubber tubing.

1 ml of the gaseous sample is now injected into the chromatograph and by suitably manipulating the attenuator all the peaks are recorded on the chart.

Measure the areas of all the peaks and calculate the percentage (*m/m*) with the help of response factor (*see* **A-2.3.4**).

A-2.3.4 *Determination of Response Factors*

a) Corresponding to each peak of the standard mixture, determine the amount of area produced by mass percent of the component.

b) Select one peak (monomethylamine) as a reference. Set its response factor (area by mass percent) equal to 1, and express all other respone factors relative to it.

A-2.3.5 *Calculation of Mass Percent of Components in Sample*:

a) For each peak, divide the measured area by the relative response factor to obtain corrected area,

b) Add up all the corrected areas and calculate each corrected area as a percent of the total corrected area. These percentages are the mass percentages of the components in the sample.

A-3. ANALYSIS OF DIMETHYLAMINE SOLUTION

A-3.0 General — The strength of the particular methylamine in solution is determined in two stages. In the first stage total alkalinity of the solution is determined by titrating against standard acid and the alkalinity is expressed in terms of percent of the particular amine. In the second stage the impurities in the solution are determined gas chromatographically and

each impurity (ammonia and amines) is expressed as the corresponding amine of which the solution is made. The sum total of these impurities is then subtracted from the total amine content to get the percent of the dimethylamine.

A-3.1 Determination of Total Alkalinity (as Dimethylamine)

A-3.1.1 *Reagents*

A-3.1.1.1 *Standard hydrochloric acid* — 1 N.

A-3.1.1.2 *Phenolphthalein indicator solution* — Dissolve 0·1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-3.1.2 *Procedure* — Take about 100 ml of water in a 250 ml conical flask and weigh. Pipette 10 ml of sample into it, keeping the tip of the pipette dipped in water while releasing the sample. Weigh it again. The difference of mass gives the mass of the sample. Titrate the contents with standard hydrochloric acid using phenolphthalein solution as indicator.

A-3.1.3 *Calculation*

$$\text{Total alkalinity } X_1 \text{ (as dimethylamine)} = \frac{V \times N \times 0{\cdot}045 \times 100}{M}$$

where

V = volume in ml of standard hydrochloric acid used in the titration with the sample solution,

N = normality of standard hydrochloric acid, and

M = mass in g of the sample taken for test.

A-3.2 Determination of Impurities by Gas Chromatographic Method

A-3.2.1 *Apparatus*

A-3.2.1.1 *Gas chromatograph* — with thermal conductivity detector (hot wire type).

A-3.2.1.2 *Potentiometric strip chart recorder* — full scale deflection 1 mV.

A-3.2.1.3 *Column* — of stainless steel or glass, 185 cm long, 4 mm internal diameter and 6 mm external diameter packed with Porapak Q (500-842 micron) coated, with a 10 percent mixture of 8·9 percent (*m/m*) tetraethylene-pentamine and 1·1 percent (*m/m*) potassium hydroxide.

A-3.2.1.4 *Syringe* — 10 micro-litre.

A-3.2.2 *Test Substances*

A-3.2.2.1 Ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water.

A-3.2.2.2 *Standard for analysis* — A standard mixture of ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water is prepared on *m/m* basis, preferably in concentration similar to that expected in the sample, taking care to see that the total vapour pressure of the mixture does not exceed 98·06 kN/m².

A-3.2.3 *Procedure*

A-3.2.3.1 The operating parameters of gas chromatograph are the following:

a) Column temperature — 90°C,

b) Injection temperature — 150°C,

c) Detector block temperature — 150°C,

d) Carrier gas — Hydrogen. Flow rate: 50 ml/min. Delivery pressure: 137·3 kN/m²,

e) Bridge current — 200 mA, and

f) Chart speed — 30 cm/h.

A-3.2.3.2 *Test procedure* — Check and adjust the gas chromatograph. Inject 1 microlitre of the standard mixture with the help of the syringe. By suitably manipulating the attenuator switch, record the peaks on the chart and measure the area of the individual peaks.

Under identical conditions, 1 microlitre of the sample is injected and peak area measurement is done for call individual peaks as in the case of standard mixture.

A-3.2.4 *Elution Order* — Elution order of the component is ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water.

A-3.2.5 *Calculation*

$$P_S = \frac{A_S \times P_{std} \times S_1}{A_{std} \times S_2}$$

where

P_s = percent by mass of the component in the sample,

P_{std} = percent by mass of the component in the standard mixture,

A_S = area of the component in the sample,

A_{std} = area of the component in the standard,

S_1 = attenuation used for sample, and

S_2 = attenuation used for standard.

A-3.2.5.1 *Calculation*

Dimethylamine content, percent by mass = $X_1 - (X_2 + X_3 + X_4)$

where

X_1 = total alkalinity (as dimethylamine), percent by mass, (*see* **A-3.1.3**);

X_2 = ammonia content (NH_3) in terms of dimethylamine, percent by mass, in the sample (*see* **A-3.2.5**) = percent $NH_3 \times \frac{45}{17}$;

X_3 = monomethylamine (MMA) content in terms of dimethylamine, percent by mass, in the sample (*see* **A-3.2.5**) = percent MMA $\times \frac{45}{31}$; and

X_4 = trimethylamine (TMA) content in terms of dimethylamine, percent by mass, in the sample (*see* **A-3.2.5**) = percent TMA $\times \frac{45}{59}$.

APPENDIX B

(*Clause* 6.1)

SAMPLING OF DIMETHYLAMINE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 The sampling instrument shall be clean and dry.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in suitable, clean, dry, airtight, metal, or dark or amber glass containers on which the material has no action.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, and the month and year of manufacture of the material.

B-1.7 Samples shall be stored in the dark.

B-2. SAMPLING INSTRUMENT

B-2.1 The following forms of sampling instrument may be used:

a) Sampling bottle or can, for taking samples from tanks or drums; and

b) Sampling tube, for taking samples from bottles or small containers.

B-2.1.1 *Sampling Bottle or Can* — consists of a weighed glass or metal container with removable stopper or top to which is attached a light chain (*see* Fig. 1). The bottle or the can is fastened to a suitable pole. For taking a sample, the bottle or the can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

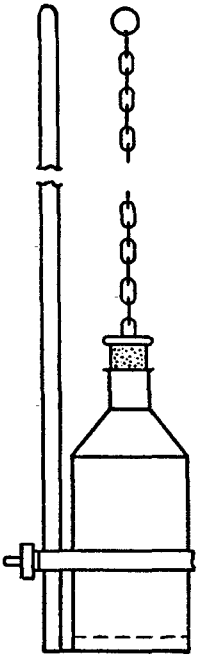

FIG. 1 SAMPLING BOTTLE OR CAN

B-2.1.2 *Sampling Tube* — made of metal or thick glass is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.

B-2.1.2.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 For Cylinders and Drums — Each cylinder or drum shall be sampled separately.

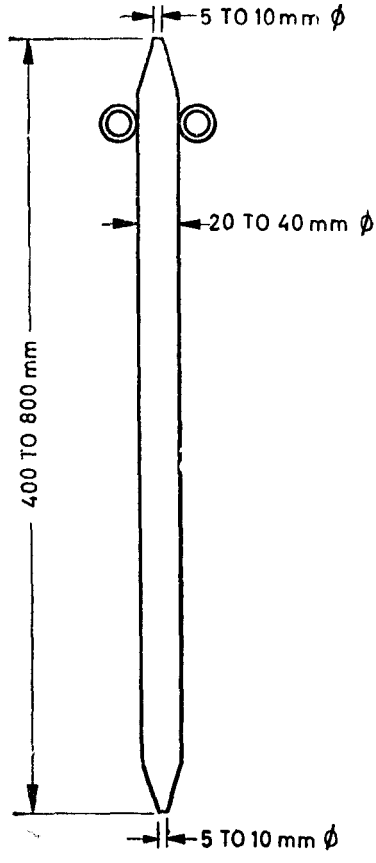

FIG. 2 SAMPLING TUBE

B-3.1.1 *Lot* — In any consignment, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 SCALE OF SAMPLING

(*Clause* B-3.2)

LOT SIZE (N)	NO. OF CONTAINERS TO BE SELECTED (n)
(1)	(2)
Up to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
111 and above	10

NOTE — Where the size of the lot is three or less, all the containers shall be sampled.

B-3.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. Reference may be made to IS : 4905-1968* for guidance to random selection procedures.

B-4. COMPOSITE SAMPLE

B-4.1 From Tanks and Drum — As far as possible, samples from a container or drum should be drawn during the operation of filling. In that case equal amounts of the material shall be collected at regular intervals so as to get a total amount of about 1 500 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different positions and depths with the sampling bottle or can after thoroughly agitating the material so as to ensure a fair amount of homogeneity. The total amount of the material about 1500 ml collected shall be thoroughly mixed and divided into 3 equal portions, one for the purchaser, another for the supplier and the third for the referee.

B-4.2 All the test samples shall be transferred to separate sample containers and sealed and labelled with full identification particulars. The referee test sample bearing the seal of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute.

B-4.3 Tests for the determination of all the requirements given in this specification shall be performed on the test sample obtained in **B-4.1**.

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to this specification if all the test results satisfy the requirements prescribed under **3** and in Table 1.

*Methods for random sampling.

INDIAN STANDARDS

ON

ORGANIC CHEMICALS (MISCELLANEOUS)

IS :

245-1970 Trichloroethylene, technical (*second revision*)
501-1976 Oxalic acid, technical and analytical reagent (*second revision*)
716-1970 Pentachlorophenol (*first revision*)
717-1969 Carbon disulphide, technical (*first revision*)
718-1977 Carbon tetrachloride (*second revision*)
869-1976 Ethylene dichloride (*second revision*)
880-1956 Tartaric acid
3321-1973 Formaldehyde solution (*first revision*)
4105-1967 Styrene (venyl benzene)
4306-1973 Hexamethylenetetramine (hexamine) (*second revision*)
4566-1968 Methylene chloride (dichloromethane), technical
5149-1977 Maleic anhydride, technical (*first revision*)
5158-1977 Phthalic anhydride, technical (*first revision*)
5254-1969 Acetanilide
5271-1969 Paraformaldehyde
5295-1969 Ethylene glycol
5296-1969 Chloroform, technical and analytical
5297-1977 Perchloroethylene (tetrachloroethylene), technical (*first revision*)
5341-1969 Benzyl chloride, technical
5464-1970 Citric acid, monohydrate
5573-1969 Ethylene oxide
5591-1969 Chlorobenzene
5592-1969 Monochloroacetic acid
5992-1969 *p*-Dichlorobenzene, technical
6393-1971 α-Phenylacetamide
6412-1971 Benzyl chloride, technical
6515-1972 Sodium pentachlorophenate, technical
6712-1972 *o*-Dichlorobenzene
6716-1972 Benzoic acid, technical
6718-1972 Phenoxyacetic acid
6768-1973 *m*-Aminophenol
6775-1973 Ethyl chloride, technical
6971-1973 2-ethyl hexan-1-OL
6972-1973 Benzotrichloride, technical
7134-1974 Diphenyl
7135-1974 Dimethyl sulphate, technical
7220-1974 Ethylenediaminetetra-acetic-acid, pure and technical
7330-1974 Methods of test for ion-exchange resins
7559-1974 Salycylic acid, technical
7618-1974 Hexachloroethane
7619-1974 Pentaerythritol
7729-1975 Sodium monochloroacetate
7901-1975 Triethanolamine, technical
7910-1975 Monoethanolamine
7911-1975 Diethanolamine
7918-1975 Diethylene glycol
8057-1976 Alpha picoline
8058-1976 Pyridine
8278-1976 Diphenylamine
8796-1977 Trimethylamine, technical
8873-1977 Monomethylamine, technical
8874-1977 Dimethylamine, technical